

# A Review of Experimental Scope, Designs and Methods from Intermediate-fast Pyrolysis of Biomass

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**Abstract**— Intermediate and fast pyrolysis (IFP) for the recovery of bio-oil from organic matter have gained the attention of researchers in their attempt to increase the contribution of renewables into the energy mix. Current research has focused on equipment configuration and variables for higher yields of the oils; methods of upgrading the oils for compatibility with existing fuel infrastructure and engines, and various tests to characterize the products or test their applicability as fuels. This paper reviews the progress in experimental work around intermediate-fast pyrolysis (hot vapour residence~1-20s; moderate to high liquid yields) in the past twelve years. The review focuses on the experimental scope, equipment used, preparation of raw materials, experimental design and characterization of bio-oils. Experimental work covering actual applications of the oils are not covered in this review paper. The feedstocks mostly researched on in IFP were rice husks, followed by pinewood, *Jatropha curcas* cake and rapeseed respectively. Most IFP studies have been done on woody biomass (over 100 different feedstocks) due to their consistency, followed by agricultural residues then herbaceous energy crops. Lignocellulosics proved to be the veteran organic feedstocks (~95% of IFP) ahead of non-lignocellulosic biomass (~5%). The most applied technologies in recent years, were fluidized bed followed by the free fall reactors. For the experimental design, most papers reviewed used the simple single parameter method, while a few used the central composite rotatable design and full factorial design methods. The characterization tests mostly conducted on the oils were the pH, viscosity, Karl Fischer titration and calorific value.

**Keywords**- *experimental, intermediate-fast, pyrolysis, methods*

## I. INTRODUCTION

Biomass refers to organic matter that originates from the process of photosynthesis. The pyrolysis of such organic matter in the form of agricultural, forestry and municipal solid wastes and intentionally grown energy crops to obtain renewable fuel, with char and gas by-products, has increasingly gained the attention of global research [1]. Pyrolysis essentially involves heating feedstock in an inert environment to obtain solid, liquid and gaseous products whose yield vary depending mainly on the conditions and equipment used [2]. The bio-oil obtained can be used for biofuels, heat, power and chemicals production, after various degrees of processing. The global drivers for intermediate-fast pyrolysis (IFP) research from a fuels viewpoint include goals to increase renewable energy contribution into the energy mix; environmental concerns; a push for more circular economies;

government policies and support; socio-economic prospects for remote and rural areas; and potential foreign exchange savings [3], [4]. On the other hand, the reason why IFP pyrolysis in particular would have such attention is due to the high crude liquid fuel yields per unit time (40 to 75%) at relatively low costs for the raw materials and operation, notwithstanding the current challenges in processing the crude oil [5]. IFP not only presents an efficient way of utilizing waste biomass, but also concentrating it into an energy dense intermediate (bio-oil) that can then be used in a number of applications. This concept of a centralized bio-refinery with a versatile source of organic raw materials and potential applications, with an easily transportable intermediate is one of the prospective highlights in IFP research [1], [5].

### A. Scope and methodology

There have been a couple of reviews for results from IFP and technologies used ([1], [5], with only one recently reviewing the operating parameters that have been used. A holistic review of experimental work covering more aspects is pertinent to cover this gap and become a one stop source for researchers desiring to carry out experimental work. The thrust of this research is to broadly review state of the art trends in lab scale and pilot plant IFP researches. The review covers experimental work, including materials studied, equipment used, preparation of raw materials (excluding their characterization), experimental design and characterization of bio-oils. It is largely a desktop study of the experimental work in IFP research of biomass in the past 12 years to establish state of the art and prospective research trends and tools. IFP covers pyrolysis with hot vapour residence times of 1-20s, moderate yields of liquids (~40%  $\approx$  char) and low gas yields (~20%) [6]. Only a representative sample of papers is chosen from the feedstock categories. The findings are reviewed discussed in the sections they are presented.

## II. EXPERIMENTAL SCOPE

This section is divided, in terms of organic materials studied, into lignocellulosic organics and non-lignocellulosic organics, where the former is distinguished by fibrous cellulose, hemicellulose and lignin constituents. Guedes et al. (2018) came up with a database of pyrolysis experimental work conducted on 174 biomass forms in the period 1984-2018 [1]. In terms of the feed stocks most used (number of

researches), the descending order was rice husks, pine wood, *Jatropha curcas* cake, palm shell and rapeseed. However, some studies, though more in number, had fewer experimental observations as shown in figure 1 for the selected best sample.

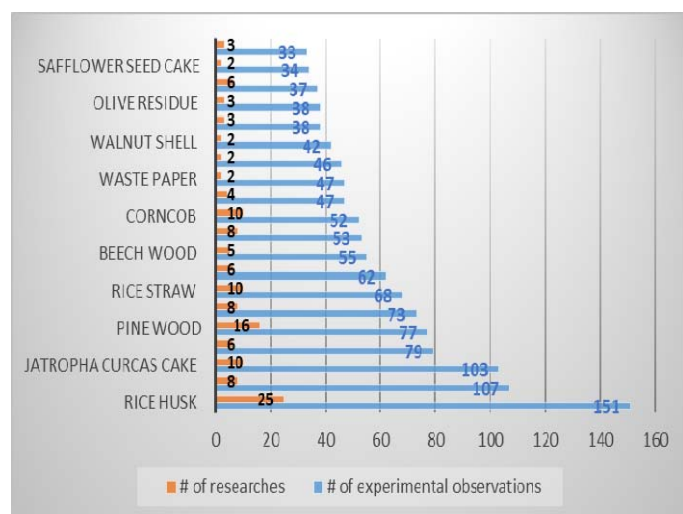


Figure 1: Biomass with highest number of researches and observations.  
Graph data adapted from [1]

The high number of researches for certain feedstock can be correlated with the abundance and availability of these waste feedstocks in areas with a good research and development (R&D) culture. For instance, on average Asia spends 0.1-4.6% of its Gross Domestic Product (GDP) on research and development and houses all the top ten rice producers globally<sup>1</sup>. Pine happens to be one of the most common woods used for lumber with the highest hectareage of specified tree species<sup>2</sup>.

#### A. Lignocellulosic organics

Just over 95% of research in IFP is on lignocellulosic matter directly obtained from plants, like agricultural and forestry residues and/or energy crops. This is followed by indirect derivatives like filter cakes from presses [1]. Authors highlight that lignocellulosic residues are the most abundant organic matter available, while energy crops are gaining popularity due to their ability to grow in marginal lands and both feedstocks have a minimal impact on food security [3], [7]. Danje (2011) observed that, although fast pyrolysis (FP) has been mostly applied to wood due to its consistency and comparability, over 100 different feedstocks have been studied [8]. Research around herbaceous grass feedstocks (energy crops) has been less frequent, while agricultural residues have enjoyed moderate attention [9]. The majority of the authors use only one feedstock and alter other variables while a few others have used a range of feedstocks for comparative analysis. Pattiya and Suttibak (2012) explored the fast pyrolysis of cassava residues from tropical and subtropical regions using a fluidized bed reactor with a hot vapour

filtration unit to obtain bio oil with low particulate levels. They argued that, while the cassava has been grown for ethanol, the rhizome and stalk are the most unutilized part of the plant with ~115million tonnes residues burned annually around the globe [10]. Although most authors point out gaps in research around their feedstocks or processes, Montoya et al. (2015) gave a more comprehensive list of the prominent research questions in current fast pyrolysis research for lignocellulosics. These include insufficient understanding of the devolatilization process, biomass decomposition kinetics and intra particle interactions between solid, gas and liquid phases. Montoya et al. (2015)'s experimental study covered the production of bio-oil from the fast pyrolysis of sugarcane bagasse from Colombia using a fluidized bed reactor [2]. Ngo, Kim and Kim (2013) discussed their findings on the fast pyrolysis of palm kernel cake using a fluidized bed reactor and focused on the experimental design and characterizing the bio-oil [11]. Several other authors compared a wider variety of lignocellulosic feedstocks, be it forestry biomass alone ([4], [12], [13]), woody biomass and agricultural residues [2], agricultural residues alone ([14], [15]), herbaceous grasses or co-pyrolysis of various biomass [16]. The highest yields for the lignocellulosics, from reviewed papers and literature, were obtained between 460 and 520°C [5]. The herbaceous grasses occupied the lower end of the range, while the woody biomass occupied the higher end due to the abundant, temperature recalcitrant lignin. Reviewed results and literature seem to suggest that woody biomass have the greatest capacity for bio-oil yields (max ~75%), followed by agricultural residues (max ~65-70%), while grasses and straws yield the least (max ~55-60%) at optimum temperatures with similar equipment [2], [5], [10]. This is partly because agricultural residues and grasses have a higher ash content, which diminish the volatiles and subsequent bio-oil yield from the condensable fraction.

#### B. Non-lignocellulosic organics

After lignocellulosics, the next most researched organic feedstocks are human and animal excreta. Guedes (2018) recorded 6 researches on sewage sludge that were carried out between 2002 and 2018 [1]. It is however surprising that their database did not capture the significant work done in poultry litter and to a much lesser extent, other animal excreta and cow dung [17]–[19]. They however, recorded the pyrolysis of fats from lamb, poultry and swine [1]. Animal excreta typically have high ash contents (up to 25 dry w/w %) compared to woody biomass (2-3%) and energy grasses (2-6%). Such excreta usually has low bio-oil yields (max~ 50%) although poultry litter can produce up to 70% yields, probably due to the catalytic effect of inorganics on the organic mix of lignocellulosics and non-lignocellulosics [20].

#### C. Technologies/Equipment used and variables explored

Montoya et al. (2015) asserted that technologies using fluidized bed (FB) and free-fall reactors are the most applied or discussed in experimental literature and industrial scale projects for FP. They give a general comment that this is due

<sup>1</sup> <http://uis.unesco.org/en/news/rd-data-release>

<sup>2</sup> <http://www.fao.org/3/y2316e/y2316e0b.htm>

to their low construction and operating costs compared to other alternatives for FP [2]. According to Bridgwater (2011), until 2011, the next most used equipment after the fluidized bed was the transported bed and circulatory fluidized bed (CFB) with 8 units commercialized and 6 in research. They did not mention free fall reactors, which evidently, had not become very popular by 2011. Bridgwater et al. (2012)'s review of FP and upgrading listed existing commercial and research FP equipment as shown in Table 1. There has not been an updated list generated until now [5].

Table 1. Existing commercial and research FP equipment by 2011 [5]

Type of equipment/technology	Number of commercial units	Number of research units
Fluidized bed	12	35
Spouted FB	1	2
Transported bed and CFB	9	5
Rotating cone	4	1
Integral catalytic pyrolysis	Not known	5
Vortex	0	1
Centrifuge reactor	0	1
Ablative	2	4
Augur/screw	9	5
Radiative-convective	0	1
Entrained flow	0	3
Microwave	>4	10
Moving bed and fixed bed	3	7
Ceramic ball down flow	0	1
Vacuum	1	None known

Considering the whole IFP range, fixed bed reactors could have lower capital and operating costs than, especially, FB. Moreover, fixed bed reactors are simpler to build and operate and require less technical expertise. This confirmed by Guedes et al. (2018), who was not particularly focused on FP as the other authors, but on IFP [1]. In this IFP range, the greatest advantage of fluidized bed reactors then, is the high yields of bio-oil compared to fixed bed reactors due to the higher rates of heat transfer for the former, a matter that all authors concur on [2], [8]. Freefall reactors have also become popular recently, with a number of researches like [15], [21] coming up since 2012; therefore Montoya et al. (2015)'s observation is credible [2]. Guedes et al. (2018) observed that from 2394 studies they reviewed, the process variables with the most investigations from a total of 12 were temperature (2379), average particle size (1648), maximum particle size (1640) and the type of pyrolysis (1528). Of all the reviewed papers, 72.8% of them investigated the effect of temperature, while only 27% and 20% studied the effect of vapour residence time and size of particles respectively on the pyrolysis outputs. This suggests that temperature is indeed the most critical parameter of the process, although it could also be due to the fact that it is relatively easier variable to manipulate. Besides the 12 process variables, there were also 12 biomass (independent) variables and 12 bio-oil (dependent) variables investigated in the researches [1].

### III. RAW MATERIAL PREPARATION

Table 3 o shows the methods used for the preparation of both lignocellulosic and non-lignocellulosic biomass.

The drying of biomass did not present any challenges across all feedstocks. However authors like Tumuluru et al. (2013) and Cai et al. (2017) have pointed out to the irregularities associated with biomass grinding and sieving, because the particles are usually broken into oblong, near-cylindrical rather than near-spherical shapes [22], [23]. This makes classification difficult and any deductions from particle size distribution (PSD) cannot use the common models associated with near spherical particles.

### IV. EXPERIMENTAL DESIGNS

Various experimental designs have been employed. Most researchers used the simple single parameter optimization method. The other design methods used by a few researchers were the central composite rotatable design (CCRD) and the Full Factorial Design (FFD) (table 2). Guedes et al. (2018) suggest that that more experimental designs that investigate the simultaneous, interactive influence of process parameters on both the yields and quality of bio-oil should be done [1].

Table 2: Experimental designs applied by various researchers

Experimental design/method	Reason it is selected	References
<i>Simple single parameter optimization</i> - one parameter is varied while the others are kept constant. Experiments usually done in doubles or triplicates. Single runs common for longer pyrolysis methods, some opting to replicate one 'centre' run to determine error.	Default method used by many authors because it is simple to follow and suitable for longer pyrolysis experiments like which may not allow for many runs over time.	[4], [9], [10], [14], [15], [21], [24]
<i>Central composite rotatable design (CCRD)</i> - A second order design method where a matrix of coded variables are set up, with number of total experiments (n) depended on a factor (k) and number of experiments at centre point ( $n_0$ ). Experimental data is fitted into a 2 <sup>nd</sup> order mathematical model then regression and statistical checks are done. Ellens & Brown (2013) use CCRD along with SAS Institute's JMP software for statistical analysis [21].	To enhance validity and objectivity of conclusions. Authors mention that it is the most popular for use in chemical engineering experimental work.	[11], [21]
<i>Two level Full Factorial Design (FFD)</i> - Comprising $2^n$ runs and $n_c$ centre runs; where $n$ is number of factors investigated and $n_c$ is the number of centre runs replicated. Replicates are used to evaluate experimental error. Results for each response variable subjected to analysis of variance (ANOVA) using Design Expert software.	Method is suitable in studying the influence of process variables and their <i>interactions</i> on the product yields (response variables). Suitable for study with few (3) variable factors- bed temperature, nitrogen flow rate and solid feed rate. Optimum point indicated by 3D curvature showing a stationary point. Other primary or secondary response variables like hydrogen yield can be studied.	[25]

## V. PRODUCT CHARACTERIZATION

The most applied characterization methods for the bio-oil were the Karl Fischer titration (for water content) and determination of pH, kinematic viscosity, stability, ash content, calorific value and composition using gas chromatograph with a mass spectrometer (GCMS) or high pressure liquid chromatography (HPLC). Of the 12 reviewed papers that characterized the products, a majority conducted the pH (83.3%), viscosity (83.3%), Karl Fischer (75%) and calorific value (66.7%) tests with the exceptions of Ngo et al (2013), Fonts et al. (2008) and Ellens & Brown (2012) who concentrated on experimental design, modelling and the effect of process variables on response variables [11], [21], [25].

The stability, ash content, GCMS and HPLC tests were done by a minority of the researches reviewed (16.7%; 25%; 8.3% and 8.3% respectively). Evidently, the majority of the researchers took interest in the physico-chemical properties of the bio-oil, which give immediate impressions on their fuel capabilities. However, GCMS and HPLC also give relevant information on chemical composition which affects fuel properties. Lira et al. (2013) are the only authors reviewed who perform an ultimate and proximate analysis of their bio-oils since their aim was to establish the effect of temperature on their quality [26]. Guedes (2018) recommend that further research be done to investigate the effect of process parameters on especially the composition, viscosity, pH and calorific value of bio-oils [1].

Table 2: Pre-treatment methods for various biomass

Biomass	Drying	Size reduction	Classification	References
Sugarcane bagasse, palm kernel cake	Bagasse is first sundried before milling to <10% w/w% moisture content (MC). Palm kernel cake milled samples thermally dried for 24h at 80°C in a drier	Bagasse ground by a hammer mill. Palm kernel cake ground using a knife mill before drying.	Sieve analysis	[2], [11]
Woody biomass, seeds and residues	Most sawdust residues seem to have achieved recommended MC during outdoor storage. Some are air or sundried for various periods.	Besides one paper reviewed were the 'as receive' samples were screened directly, the rest had to be ground. Some authors use hammer mill only [4], others subsequently use the knife mill [21]. For FB, seeds had to be further ground, but in some cases they were used as received depending with reactor.	Sieve analysis. Ellens & Brown (2012) mention use of 0.64cm screen. Reza et al. (2019) & Lira et al. (2013) use particles below 0.25mm & 2mm, while other authors classify into multiple size ranges.	[4], [21], [26], [27]
Herbaceous grasses and energy crops e.g. miscanthus	Oven dried at 103 °C for 24 hours;	Forage chopper used for grasses; hammer mill for the silage.	Sieve analysis	[9], [16]
Crop residues e.g. cassava, maize, wheat,	Sirijanusorn et al. (2013) first sun dry to 15% MC, then oven dry at 105°C for 48hrs until 'bone dry' [14]. Materials in other researches are either oven or sun dried.	Rice straw cut and milled to diameter ~1mm-. Equipment for corn cobs- hammer mill.	Sieve analysis	[8], [10], [14], [15]
Excreta (sewage, poultry litter, pig manure, cow dung)	Sewage anaerobically digested and thermally dried [25]; Cattle manure air dried to 10.14 ± 0.5 wt% in greenhouses [17]	Broiler litter samples milled in Retsch cross-beater mill to <1 mm and pelletized [18]; Other litter knife milled [19]; Cattle manure 'crushed' [17].	Sieve analysis	[17]–[19].

## VI. CONCLUSION

The majority of IFP research has been done on abundantly available waste feedstocks like rice husks and pine wood residues with an ecological justification for their valorization. It appears that research has been skewed towards certain feedstocks, technologies, parameters and experimental designs with a natural preference for easier or cheaper research avenues. For instance, there could be abundant feedstocks in areas with low promotions on R&D, while other pertinent feedstock like invasive species and excreta have received

much less attention compared to the frequently explored lignocellulosic categories. Such feedstock research gaps can be closed by encouraging researchers through various incentives and grants for those in developing regions. Research should also shift from conventional studies of the effect of variables like temperature and particle size on the yields to look into time technical research gaps highlighted by Montoya (2015) [2]. This include understanding of the devolatilization process, biomass decomposition kinetics and intra particle interactions between solid, gas and liquid phases. Armed with the knowledge of high oil yielding species and the

properties of the oils, more research should also now focus on simple and rigorous upgrading techniques of the bio-oil to answer various immediate societal needs like energy poverty in developing nations. Techno-economic assessments on the sustainability of using the upgraded oils directly or with engines in various power options can then be established. To reduce bio-oil upgrading costs, it will also be important to investigate the effect of process parameters on the composition, viscosity, pH and calorific value of bio-oils, so that optimum parameters are used during the pyrolysis process to yield bio oils with desirable qualities. The fluidized bed and freefall reactors could have been the most used equipment, however, cost-benefit analysis have to be done for bigger scales, in comparison with the simpler fixed bed designs, which will have smaller oil yields but lower capital and maintenance costs.

#### ACKNOWLEDGEMENTS

The authors would like to acknowledge Botswana International University of Science and Technology and the University of Johannesburg for supporting the studies as well as conference attendance.

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